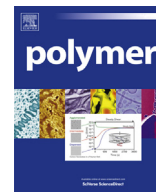




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## Dispersability of multiwalled carbon nanotubes in polycarbonate-chloroform solutions

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### ABSTRACT

The dispersion of commercial multiwalled carbon nanotubes (MWCNTs, Nanocyl™ NC7000) in chloroform and in polycarbonate (PC)-chloroform solutions was investigated by variation of the polymer concentration, MWCNT amount and sonication time and compared with PC/MWCNT composites, which were processed by melt mixing, subsequently dissolved in chloroform and dispersed via sonication under the same conditions. The sedimentation behaviour was characterised under centrifugal forces using a LUMiSizer® separation analyser. The space and time resolved extinction profiles as a measure of the stability of the dispersion and the particle size distribution were evaluated. Sonication up to 5 min gradually increases the amount of dispersed particles in the solutions. A significant improvement of the MWCNT dispersion in chloroform was achieved by the addition of PC indicating the mechanism of polymer chain wrapping around the MWCNTs. In dispersions of melt mixed PC/MWCNT composites the dispersion of MWCNTs is significantly enhanced already at a low sonication time of only 0.5 min due to very efficient polymer wrapping during the melt mixing process. However, the best dispersion quality does not lead to the highest electrical conductivity of thin composite films made of these PC/MWCNT dispersions.

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### 1. Introduction

For the development of electrically conductive carbon nanotube (CNT) filled polymer nanocomposites a sufficient dispersion of the CNTs in the polymer matrix is essential. A good quality of dispersion strongly depends on various parameters like the type of CNT and its geometrical dimensions, the surface properties of CNTs and polymer, the matrix viscosity and the dispersion method, to name but a few. The dispersion of CNTs in thermoplastic polymers via melt mixing and the significant impact of melt mixing parameters like temperature, screw speed or mixing time on the dispersion of CNT and therefore on the electrical properties of the composites have been intensively studied [1–7]. Further the aspects of polymer viscosity [8,9] and choice of CNT type were discussed by several authors [8,10–13]. To develop thin films of electrically conducting polymer/CNT nanocomposites processing from solution like solution casting, dip coating or spin coating is required. The dispersion of CNTs in organic solvents is supported by sonication, a process which generates cavitation to break the CNT agglomerates, instead of shear forces generated by the mixing elements during melt

mixing. Parameters like sonication time, frequency and amplitude significantly influence the degree of CNT dispersion. For many applications surface modification of CNTs is an essential tool to effectively disperse CNTs in solvents and polymers [14].

A number of studies have been published within the last 10 years, characterising the dispersability of CNTs in organic solvents in correlation with their surface properties, considering the solubility parameters of nanotubes and solvents. Up to now, these efforts have mainly concentrated on dispersions with singlewalled carbon nanotubes (SWCNTs) [15–18]. The dispersion of multiwalled CNTs (MWCNTs) in various organic solvents was discussed by Detriche et al. [19,20]. Few studies focus on the dispersion of CNTs in polymer solutions [21,22]. Some authors discussed polymer wrapping at CNTs, which was specifically used to improve the dispersion of CNTs in water [23] or organic solvents [24]. Theoretical studies reported the effective wrapping of polymers containing aromatic phenyl rings due to a high binding energy of the polymer resulting in high interfacial adhesion with SWCNTs [25] and postulated a dependency of the interaction strength of the CNT with the polymer on the number of repeating units in the polymer [26]. The group of Szleifer and Yerushalmi-Rozen suggested the decoration of SWCNTs with end-tethered polymers as the entropic repulsion among the tethered chains generates a free energy barrier that prevents the SWCNTs from approaching [27,28].

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The quality of the CNT dispersion in a polymer solution strongly depends on the type of polymer and solvent which are used, the polymer concentration and CNT content in the solvent, and the ultrasonic treatment of the dispersion.

In the present work the dispersion of non-functionalized MWCNTs in chloroform and polycarbonate(PC)-chloroform solutions was investigated and compared with PC/MWCNT composites, which were pre-treated via melt mixing and subsequently dissolved in chloroform. This pre-treatment was performed to further improve the MWCNT dispersion and consequently enhance the electrical conductivity of the prepared films. To qualify the dispersion stability and the particle size distribution of the MWCNTs in the solutions a centrifugal separation analysis was performed. This well-established method [29,30] was already applied by Krause et al. [31] to investigate the dispersability of different types of CNTs in aqueous surfactant solutions using a LUMiSizer® centrifugal separation analyser. Thereby a correlation between the stability of the aqueous MWCNT dispersions and the dispersability of the nanotubes in polymer melts of polyamide 6.6 was found. A conformance of the sedimentation behaviour of aqueous MWCNT dispersions with the MWCNT dispersion in polycarbonate melts was also shown by Pegel et al. [10]. A further study focussed on the dispersability of four different commercially available MWCNT materials in aqueous surfactant solutions as a function of ultrasonic treatment time [32]. In that work best dispersabilities were found for MWCNTs of type Nanocyl™ NC7000 and Future Carbon CNT-MW. During the last years centrifugal analysis has become a favoured tool to study various types of CNT dispersions [33–35].

## 2. Experimental

An overview about the experimental study is given in Fig. 1. The experimental details are described in the following sections.

### 2.1. Materials

Commercially available multiwalled carbon nanotubes NC7000 from Nanocyl™ (Sambreville, Belgium) were used for this study.

They were produced in an industrial large-scale catalytic vapour deposition process and have an average diameter of 10 nm, an average length of 1.3  $\mu\text{m}$  [12], a carbon purity of 90% and a surface area of 250–300  $\text{m}^2/\text{g}$  [36]. As found in former studies NC7000 are characterised by a good dispersability in various thermoplastic polymer melts [8,10] and in aqueous dispersions [31,32] compared to other types of MWCNTs. The polymer used in this study was a medium viscosity grade PC Makrolon® 2600 from Bayer MaterialScience AG, Germany. The MWCNTs and PC were dried at 120 °C for 4 h in a vacuum oven before processing.

### 2.2. Preparation of MWCNT-polymer-solvent-dispersions

Three different types of dispersion were prepared (see Table 1). For the first one, MWCNTs NC7000 were added to polycarbonate-chloroform solutions with the polymer concentration in the chloroform varying between 0.25, 0.5 and 1 wt%. The MWCNT content was fixed to 1 wt% referred to the added polymer, i.e. the MWCNT amount in the solution was increasing from 0.037 g/l to 0.74 g/l to 0.148 g/l with increasing polymer concentration. The dispersions were named as *PCCL dispersions*. The attached number refers to the polymer concentration, e.g. *PCCL025* contains 0.25 wt% of PC in chloroform, *PCCL050* contains 0.5 wt% and *PCCL100* contains 1 wt% of PC in the solvent. As reference, equal MWCNT amounts were added to pure chloroform under the same conditions. These dispersions are named as *CL dispersions*. To remain consistent in the notation of the samples the numbers 025, 050 and 100 are also used for the *CL dispersions* but due to absence of polymer they refer to the MWCNT content which is equal to the corresponding *PCCL dispersions*. For the third type of dispersion, melt mixed PC/MWCNT composites with 1 wt% of MWCNT were solved in chloroform in an analogous manner. The compounding process is described in Section 2.3. The dispersions made from melt mixed PC/MWCNT composites are called as *PCcomp dispersions*.

The amount of solvent used for the dispersion of the MWCNTs was fixed to 10 ml. To disperse the MWCNTs in the solvents and in the polymer solutions sonication was applied at room temperature for 0.5, 1, 2, 5 and 10 min using an UP200S processor (Hielscher Ultrasonics GmbH, Teltow, Germany) with a maximum frequency

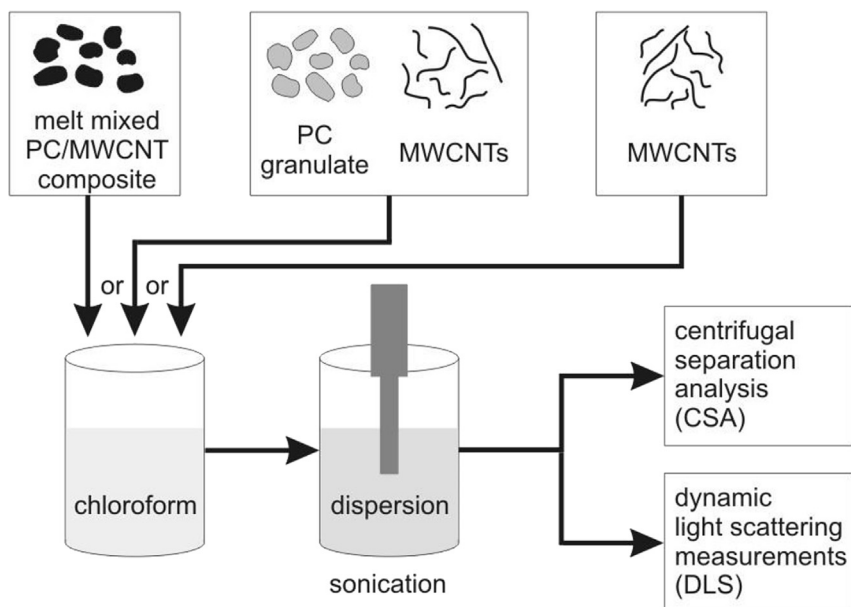


Fig. 1. Scheme of the preparation and analysis of the studied MWCNT dispersions.

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